U.S. DEPARTMENT OF AGRICULTURE GRAIN INSPECTION, PACKERS AND STOCKYARDS ADMINISTRATION FEDERAL GRAIN INSPECTION SERVICE STOP 3630 WASHINGTON, D.C. 20090-3630 NIRT HANDBOOK CHAPTER 1 11-22-99

Near-Infrared Transmittance Testing

CHAPTER 1

INTRODUCTION

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Near-Infrared Transmittance Testing

INTRODUCTION

1.1 PURPOSE

This handbook establishes procedures for determining and certifying official protein content in wheat, official protein and oil content in soybeans, official protein, oil, and starch content in corn, monitoring the accuracy of official results, and maintaining near-infrared transmittance (NIRT) equipment accuracy.

1.2 SCOPE

Testing wheat for protein content, soybeans for protein and oil content, and corn for protein, oil, and starch content as "official criteria" is authorized under Section 7(b) of the United States Grain Standards Act (USGSA), as amended. Sections 800.125 and 800.135 of the USGSA permits a review inspection on either official grade/factors or official criteria. When requested, a review inspection for official grade or official factors and official criteria may be handled separately even though both sets of results are reported on the same certificate. All official protein, oil, and starch analysis under the USGSA shall be performed in accordance with procedures prescribed in this handbook and the instrument manual and must be performed only by authorized or licensed employees of the Federal Grain Inspection Service (FGIS) or delegated/designated agencies.

1.3 **DEFINITIONS**

- a. **Baseline** Known protein, oil, and starch values determined by the Inspection Systems Engineering Branch (ISE), Technical Services Division (TSD), for the Standard Reference Sample (SRS) sets.
- b. **Bias** The average difference between NIRT results and the baseline values assigned by ISE to the SRS. To "bias" an NIRT instrument, adjust the instrument's intercept constant by the negative of the amount of the calculated bias.
- c. **Calibration Constants** The instrument requires a calibration disk containing the necessary calibration constants. The NIRT instrument uses these numerical values to calculate the protein content for the class of wheat being tested, the protein and oil content of soybeans, and the protein, oil, and starch content of corn.

- d. **Calibration Disk** A Tecator Infratec compatible floppy disk where the NIRT calibration constants, instrument constants, and the instrument operating system are magnetically stored.
- e. **Check Samples** Samples distributed to official inspection points by ISE for the purpose of comparing field results to ISE results.
- f. **Coarse Foreign Material** Foreign material found in soybean samples that consists of the following:
 - (1) Whole kernels of corn. Whole kernels of corn are kernels with one-fourth or less of the kernel removed.
 - (2) Cockleburs.
 - (3) Sticks meeting the following criteria:
 - (a) Approximately 1 inch or more in length.
 - (b) Approximately ½ inch or more with a thickness of 5/32 of an inch (width of the largest soybean slotted sieve).
 - (4) Pods (one-half pod or more). If pods contain soybeans, remove the soybeans and return to sample.
 - (5) Other coarse foreign material may include, but is not limited to, corn cobs, large feed pellets, pieces of dirt larger than soybeans, sweet corn, and edible beans that are generally larger than soybeans.
- g. **Collaborative Study** A study designed to compare constituent values determined by different laboratories.
- h. **Combustion Method** A chemical analysis used to determine the percent nitrogen in a sample using the AOAC International Method 992.23.
- i. **Constituent** Compounds for which an analysis is made in a product (i.e., protein in wheat, protein and oil in soybeans, protein, oil, and starch in corn).
- j. **Correlation** The interdependency of one variable on another (i.e., solvent oil extraction and NIRT oil).

k. **Instrument Constants** - Several constants (some of which are different for each instrument) entered on the instrument's calibration disk prior to using the disk for official determinations.

The instrument constants that must be entered for each unit include "O" and "P" constants (which characterize the instrument's optical components) and the slope and intercept constants that adjust each instrument for optimum agreement with the standard reference method. No other constants shall be altered by the operator.

Disks containing calibration constants are no longer interchangeable among NIRT units after instrument constants are entered. Each NIRT unit must be used with its own calibration disk.

- 1. **Monitor Samples** Samples selected by a specified process from the officially inspected samples analyzed during a period of time. The official results on these samples are compared to the results obtained by a monitoring office to detect developing protein inaccuracies in wheat, protein and oil inaccuracies in soybeans, and protein, oil, and starch inaccuracies in corn.
- m. **Near-Infrared Transmittance (NIRT) Determination** A spectrophotometric determination of a sample's constituents by measuring the amount of light transmitted through a sample at specific wavelengths in the near-infrared region of the spectrum.
- n. **Oil** Lipids (oils and fats) that are liquid at room temperature.
- o. **Pathlength -** With the sample cell installed in the instrument, it is the distance between the two glass windows on opposite sides of the grain stream. One of these windows is on the detector module and the other is on the removable sample cell.
- p. **Polarimetry -** A technique that measures the rotation of the plane of polarized light **or** the degree of polarized light passing through an optical sample to determine the starch content using the Corn Refiners Association Method A-20.
- q. **Protein** A naturally occurring complex combination of amino acids joined by peptide bonds that contain the elements carbon; hydrogen; nitrogen; oxygen; sulphur; and, to a lesser degree, other elements.

- r. **Sample Transport Module -** An accessory to the Infratec 1225/1226/1227 Grain Analyzer used in place of the hopper, sample cell, and brush wheel, to analyze small sample portions.
- s. **Solvent Extraction** A process that uses a non-polar solvent such as petroleum ether to extract the oil from a sample using the FGIS solvent oil extraction method.
- t. **Specified Service Point** A city, town, or other location where official services are performed by an official agency or FGIS personnel.
- u. **Standard Reference Method** A standardized chemical method used to determine a constituent value for a sample. FGIS standard reference methods are used to develop the calibration constants used with NIRT instruments for each class of wheat, and for soybeans and corn.
- v. **Standard Reference Samples (SRS)** A set of samples with established protein values for each class of wheat, protein and oil values for soybeans, and protein, oil, and starch values for corn which are used to maintain NIRT instrument accuracy.
- w. **Standard Slope Settings -** Average slope value used by all instruments within the official system. It is based on the individual slope values for instruments maintained by the TSD and covers grain from multiple crop years.
- x. **Starch** Any of a group of polysaccharides composed of long-chain polymer of glucose in the form of amylose and amylopectin. It is the chief storage form of energy reserve (carbohydrates) in plants.

1.4 RESPONSIBILITIES

The general responsibilities for the wheat protein, soybean protein and oil testing, and corn protein, oil, and starch programs are as follows:

- a. Responsibilities of the Inspection Systems Engineering Branch (ISE).
 - (1) Maintain the FGIS National Standard NIRT instruments.
 - (2) Develop, maintain, and implement the calibrations for official NIRT instruments. This includes developing the standard slope settings associated with an updated calibration and issuing the standard slope settings used by official NIRT instruments.

- (3) Establish the official SRS protein contents for each class of wheat, protein and oil contents for soybeans, and protein, oil, and starch contents for corn.
- (4) Develop and distribute SRS sets to FGIS field offices and official agencies.
- (5) Prepare and distribute check samples (biannual for soybeans, annual for corn) to monitor the accuracy of official testing locations providing soybean protein and oil, or corn protein, oil, and starch testing service.
- (6) When necessary, review NIRT procedures at FGIS field offices and official agencies.
- (7) Monitor the accuracy of official wheat protein, soybean protein and oil, and corn protein, oil, and starch testing locations.
- (8) Initiate, conduct, and report collaborative and special studies as needed.
- (9) Provide technical support and training to official inspection personnel in matters relating to NIRT determinations, maintenance, service, and repair.
- b. Responsibilities of the Board of Appeals and Review (BAR).
 - (1) Provide maintenance, service, and repair for federally owned instruments.
 - (2) Coordinate requests for Board appeal inspection services.
 - (3) As applicable, issue certificates and assess fees for Board appeal inspection services.
- c. <u>Analytical, Reference, and Testing Services Branch (ARTS)</u>. Maintain the FGIS standard reference laboratories. Establish the official wheat protein content, soybean protein and oil contents, and corn protein, oil, and starch contents for calibration samples and slope samples.
- d. Responsibilities of FGIS Field Office Managers.
 - (1) Select an NIRT coordinator to serve as the primary contact within the respective circuit and to ISE.

- (2) Perform original, reinspection, and appeal wheat protein, soybean protein and oil, and corn protein, oil, and starch inspection services as applicable.
- (3) Retrieve and distribute the wheat protein control charts and collaborate with ISE in monitoring specified service points within the circuit.
- (4) Routinely review NIRT procedures in the respective area of responsibility.
- (5) When necessary, inform ISE of problems detected in respective areas of responsibility and initiate appropriate corrective action.
- (6) Provide technical support and training to official inspection personnel.
- (7) Assist ISE in conducting collaborative and/or special studies.
- (8) Assure that official agencies select and forward wheat protein monitoring samples to the appropriate field office and/or ISE.
- (9) Mail file samples for Board appeal inspection services to the BAR using a red mailing tag. Write the words "Protein Board Appeal" in the "Remarks" section of the grain sample ticket and on the back of the mailing tag.
- (10) On a case-by-case basis and with the approval of the Director, Field Management Division, provide original wheat protein, soybean protein and oil, and corn protein, oil, and starch testing service in areas where agencies are unable to arrange for adequate services (see Section 1.4e(2)).
- (11) Send soybean and corn samples, upon request, to ISE for use in producing check samples and evaluating calibration performance.
- (12) Test soybean and corn check samples and submit results to ISE within 10 days of receipt.

e. <u>Responsibilities of Agency Managers</u>.

(1) Coordinate and maintain a wheat protein, and/or soybean protein and oil, and/or corn protein, oil, and starch testing program in assigned geographic areas.

- (2) Perform original and reinspection NIRT wheat protein, and/or soybean protein and oil, and/or corn protein, oil, and starch inspection services and forward file samples for appeal to the FGIS field office or ISE. In some instances, the demand for the wheat protein, soybean protein and oil, or corn protein, oil, and starch testing service may not warrant the purchase of NIRT equipment by an agency. If this occurs, the agency must survey industry representatives, determine the need for service, notify FGIS of their findings, and locate a mutually agreeable agency to provide the service. If an agency cannot arrange for adequate service, FGIS will determine on a case-by-case basis whether FGIS will provide original wheat protein, and/or soybean protein and oil, and/or corn protein, oil, and starch testing service.
- (3) Select and forward wheat protein monitoring samples to the appropriate FGIS field office and/or ISE.
- (4) Test soybean and corn check samples and submit results to ISE within 10 days of receipt.
- (5) Routinely review the NIRT operator's procedures at testing sites within the assigned geographic area.
- (6) Provide technical support and training to NIRT technicians.
- (7) Assist ISE/FGIS field offices in conducting collaborative and special studies.
- (8) Notify the monitoring FGIS field office concerning NIRT related problems and initiate follow-up action.
- (9) Maintain complete work records.

1.5 DISCLAIMER CLAUSE

The mention of firm names or trade products does not imply that they are endorsed or recommended by the U.S. Department of Agriculture over other firms or similar products not mentioned.